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The invention relates to a method of manufacturing a polymer-dispersed liquid crystal cell, in which method a mixture, which predominantly comprises a liquid crystalline material as well as reactive monomers and a photoinitiator, is sandwiched between two substrates, which are provided with an electrode layer, whereafter the mixture is  
 5 polymerized under the influence of radiation. The invention also relates to a polymerizable mixture which can suitably be used in a polymer-dispersed liquid crystal cell as well as on a display device comprising such a cell.

Polymer-dispersed liquid crystal cells (abbr. PDLC cells) are increasingly being used in electro-optic devices, such as display devices, optical projectors and electrically  
 10 drivable optical shutters. The optically active material of these cells is formed by liquid crystalline material which is dispersed in a matrix of a polymerized material. Such a material is referred to as a polymer-dispersed liquid crystalline material (abbr. PDLC material). This material is customarily prepared by providing a mixture of a liquid crystalline material (70-95% by weight), reactive monomers (5-30% by weight) and at least one photoinitiator, in the  
 15 form of a layer, between two substrates of a cell and, subsequently, polymerizing this layer under the influence of radiation. During polymerization, phase-separation occurs, which leads to the formation of the desired optically active layer of polymer-dispersed liquid crystalline material. This layer can be switched between an optically transparent state (in the presence of a field) and an optically scattering or translucent state (in the absence of a field) by means of an  
 20 electric field.

A method of the type mentioned in the opening paragraph is known per se, for example, from European Patent publication EP-A 575.791. More particularly, in examples 5-18 of said publication, a description is given of a prepolymer of reactive monomers composed of 2-ethylhexylacrylate (EHA) as well as one or two polyfunctional monomers. One  
 25 part by weight of this prepolymer is mixed with four parts by weight of a non-reactive liquid crystalline material. Also a small quantity of a photoinitiator is added to this mixture. The resultant polymerizable mixture is subsequently provided between two substrates and polymerized by means of UV light so as to form an optically active layer.

The known method has an important drawback. It has been found that the

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electro-optical response of the PDLC cells thus manufactured is not uniform at all parts of the surface of the cell. For example, the switching voltage necessary to switch from transparent to scattering, and vice versa, is found to be different for different parts made of PDLC material. It has further been found that the electro-optical properties of the PDLC material are

5 insufficiently stable with respect to time. Life tests show that these properties deteriorate relatively rapidly. For example, the hysteresis and the switching voltage increase rapidly.

It is an object of the invention to overcome the above-mentioned disadvantage. The invention more particularly aims at providing a method of manufacturing PDLC cells which exhibit a uniform electro-optical response, which is stable with respect to

10 time. The PDLC cells manufactured by means of the inventive method should have a relatively low switching voltage, preferably, of approximately 6 V or less as well as a relatively low hysteresis, preferably, of approximately 3 % or less. Another object of the invention is to provide a polymerizable mixture which is stable with respect to time and which can suitably be used in the method in accordance with the invention. The invention should also provide a

15 display device having an improved PDLC cell.

These and other objects of the invention are achieved by a method of the type mentioned in the opening paragraph, which is characterized, in accordance with the invention, in that the mixture comprises two types of non-volatile, reactive monomers, the first type of monomer being readily miscible with the liquid crystalline material and the second type

20 of monomer being poorly miscible with said liquid crystalline material.

The invention is based on the insight that in the case of the known cells a non-uniform electro-optical response is obtained because the composition of the PDLC material is not the same everywhere. This is attributed to the presence of EHA in the known polymerizable mixture. This compound has a relatively great volatility. During filling of the

25 cell, this compound evaporates, which leads to concentration differences in the filled cell. This results in a non-uniform electro-optical response in the known cell. EHA exhibits the greatest volatility problems if the cells are filled under the influence of a reduced pressure.

It has been found that the problem cannot be solved by simply replacing the volatile EHA with a single, non-volatile acrylate compound having approximately the same

30 molecular mass. The replacement of EHA of the known polymerizable mixture by a non-volatile, higher alkylacrylate, such as decylacrylate (DA) yields poor results. Various electro-optical properties, such as the switching voltages and the hysteresis of the switching curve, of a cell comprising such a polymerized mixture turn out to be considerably worse than those of the known cell comprising the EHA-containing mixture. It is noted that the term "non-volatile



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The invention also relates to a polymerizable mixture which can suitably be used in a polymer-dispersed liquid crystal cell, and which comprises reactive monomers and a photoinitiator. In accordance with the invention, this mixture comprises two types of non-volatile, reactive monomers, the first type of monomer being readily miscible with liquid crystalline material and the second type of monomer being poorly miscible with liquid crystalline material. PDLC cells comprising this mixture exhibit good electro-optical properties, such as, in particular, a uniform electro-optical response.

A preferred embodiment of the polymerizable mixture is characterized in that the first type of monomer is an ethoxylated alkyl-phenolacrylate whose alkyl group comprises at least five C-atoms, and in that the second type of monomer is an alkylacrylate whose alkyl group comprises at least 8 and maximally 18 C-atoms. In this connection, good results have been achieved with a mixture in which the quantity of each of the two types of monomers is at least 20% by weight, calculated with respect to the overall quantity of both types of monomers. Preferably, the ratio between both types of monomers is approximately 1:2. The polymerizable mixture is optimally suitable for use in a PDLC cell if 70-90% by weight of a liquid crystalline material of a customary type has been added. The polymerizable mixture thus obtained can be directly used to fill PDLC cells.

The invention also relates to a display device comprising a polymer-dispersed liquid crystal cell. In this case, the electrode layers of the substrates of the cell are constructed so as to form rows and columns, each row or column being individually drivable. The rows of one substrate and the columns of the other substrate are oriented so as to extend at right angles to each other. The presence of the matrix of electrodes formed by said columns and rows enables pixels of the PDLC material of the display device to be driven locally by means of an electric voltage. Preferably, each one of the pixels is provided with a solid-state switch in the form of a thin-film transistor or a thin-film diode. By virtue thereof, it becomes possible to form images. The PDLC cells manufactured in accordance with the inventive method can very suitably be used in such a display device.

These and other aspects of the invention will be apparent from and elucidated with reference to the embodiments described hereinafter.

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In the drawings:

Fig. 1 is a schematic, sectional view of a PDLC cell,

Fig. 2 shows the electro-optical curve of a PDLC cell,





example, it was found that the voltage necessary to switch from transparent to scattering, and conversely, was dependent on the location and hence not the same throughout the surface of the optically active layer of the cell. This phenomenon is ascribed to the so-called "compositional drift", which can occur as a result of evaporation of EHA during filling of the cell. When the cell is filled by means of capillary action, said evaporation occurs to a lesser degree. The cells of the second series also exhibited ring-shaped structures around the filling hole. However, these structures were less clearly visible than those of the vacuum-filled cells.

#### Experiments in accordance with the invention.

10 In a subsequent experiment, the EHA of the polymerizable mixture PN393 was replaced by a mixture of 37.5 parts by weight of ethoxylated nonyl-phenolacrylate (ENPA, see Fig. 3) and 62.5 parts by weight of tridecylacrylate (TDA, see Fig. 3). This mixture is referred to as PN393'. A quantity of 20 parts by weight of this polymerizable mixture were mixed with 80 parts by weight of the liquid crystalline material TL205 (Merck).

15 In a first series of cells, the mixture thus obtained was introduced into the PDLC cells via capillary-filling, and, in a second series, via vacuum-filling, whereafter said mixture was polymerized under the above-mentioned conditions.

Visual inspection of the PDLC cells manufactured in accordance with the invention revealed that there were no ring-shaped structures around the filling opening. In the case of both the vacuum-filled cells and the capillary-filled cells, said ring-shaped structures were absent.

Table 1 lists seven different PDLC cells, which are filled, either via vacuum-filling or capillary action, with one of the two above-described mixtures, i.e. of PN393 or PN393'. To determine the stability of the mixtures, a number of the cells were subjected to an accelerated life test after the polymerization process ("aftertreatment"). Table 2 lists some electro-optical properties of these cells, i.e. the value of the switching voltages V10 and V90 as well as the hysteresis (%). These properties were measured immediately after the manufacture of the cells (cells 1, 3 and 5) or after the cells had been subjected to life tests (cells 2, 4, 6 and 7).

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Table 1

Cell	Mixture	Filling method	Aftertreatment
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1.	PN393/TL205	Capillary	--
2.	PN393/TL205	Capillary	5 min, 90°C
3.	PN393'/TL205	Capillary	--
4.	PN393'/TL205	Capillary	60 min, 90°C
5 5.	PN393'/TL205	Vacuum	--
6.	PN393'/TL205	Vacuum	60 min, 100°C
7.	PN393'/TL205	Vacuum	960 min, 100°C

Table 2

10	Cell	1	2	3	4	5	6	7
	V10 (mV)	5.0	5.7	3.3	3.6	3.1	3.2	3.2
	V90 (mV)	8.8	10.6	6.0	6.4	5.9	6.0	6.1
	hysteresis(%)	3.0	7.1	2.6	4.4	2.9	2.9	2.8

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The Tables show that the cells comprising the polymerized mixtures in accordance with the invention exhibit a substantially lower V90 value as well as a substantially higher stability than cells comprising the known mixture. A comparison between the cells 1 and 2 (not in accordance with the invention) shows that after a life test of 5 minutes at 90 °C the hysteresis of the known mixture has already more than doubled.

The hysteresis of cells comprising the mixtures in accordance with the invention increases less than the hysteresis of cells comprising the known mixture. The hysteresis of the mixture in accordance with the invention remains substantially constant if it has been introduced into the PDLC cell via vacuum-filling. Cells which are filled in this manner also demonstrate the lowest hysteresis and the greatest stability.

A comparison of the cells 1 and 2 shows that the V10 and V90 values of the known material increase substantially after a short treatment at an elevated temperature. The cells filled by means of the method in accordance with the invention prove to be much more stable in this respect. The stablest cells are those which are vacuum-filled with the mixture in accordance with the invention.

In a number of further experiments, the ratio in which the two non-volatile reactive monomers occur in the polymerizable mixture was varied. In this case, the two non-volatile reactive monomers include the above-mentioned ENPA with one of the following alkylacrylates: decylacrylate (DA), dodecylacrylate (DDA), tridecylacrylate (TDA) or

octodecylacrylate (ODA). The chemical structural formulas of these compounds are shown in Fig. 3. ENPA is a monomer which is very readily miscible with customary liquid crystalline materials, such as PN393. The above-mentioned alkylacrylates, however, are poorly (i.e. incompletely) miscible with customary liquid crystalline materials.

5 A quantity of 80 parts by weight of TL205 (Merck) were added to 20 parts by weight of the polymerizable mixtures comprising one of the above-mentioned combinations of non-volatile monomers. The mixtures thus formed were introduced into PDLC cells at a reduced pressure. After polymerization of the mixture under the above-mentioned conditions, the hysteresis as well as the V90 or the V50 value of the cells were determined. The measured  
10 values are indicated in the graphs of Fig. 4.

Fig. 4A-C show that the lowest values for the hysteresis and for V90 or V50 are obtained with mixtures comprising at least 20% by weight of one of the two non-volatile reactive monomers. If use is made of mixtures comprising less than 20% by volume of one of the two monofunctional monomers, either the hysteresis or the V90 or V50 values  
15 demonstrate an unacceptably large increase. The lowest values are achieved at mixing ratios of the non-volatile monomers of approximately 1:2.

The invention provides a method of filling a PDLC cell, a polymerizable mixture suitable for this purpose as well as a display device provided with such a PDLC cell. The mixture in accordance with the invention comprises two types of non-volatile reactive  
20 monomers, the first type of monomer being readily miscible with liquid crystalline material and the second type of monomer being poorly miscible with the liquid crystalline material. Such mixtures prove to be very stable. In addition, when such mixtures are used in cells, problems regarding compositional drift do not occur. Cells in which the inventive mixture is used demonstrate a relatively low hysteresis as well as a relatively low switching voltage. By  
25 virtue thereof, it is very attractive to use these cells in a display device.

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